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HEAT TREATMENT FOR IMPROVED STRESS-CORROSION RESISTANCE OF 17-7 PH STAINLESS STEEL

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ABSTRACT

This paper discusses stress-corrosion tests given to 17-7 ph stainless steel—previously subjected to different heat treatments—to fit it for the requirements of rocket-driven spacecraft. Two commercial treatments and one experimental treatment are described, and illustrated by micrographs.

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INTRODUCTION

The advent of rocket propulsion, with a concomitant increase in the requirements of service life and reliability, has given a new importance to the compatibility of metallic parts with liquid propellants. The spacecraft designer must now concern himself with the possibility of propellant decomposition, metal-oxidizer reactivity, and material degradation due to corrosion.

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The stainless steels that can be hardened by semi-austentic precipitation (e.g., ph 15-7 Mo, AM 350, and 17-7 ph—to name only a few) have been considered for application to reaction motor and support equipment—on the grounds of their promising corrosion resistance, good fabricality, and high-strength properties. The 17-7 ph alloy is considered acceptable (Reference 1), but it has several times shown a tendency to crack under stress-corrosion (as shown in Figure 1) when exposed to a nitrogen-tetroxide/hydrazine system (N_2O_4 - MMH). In an attempt to reduce, if not

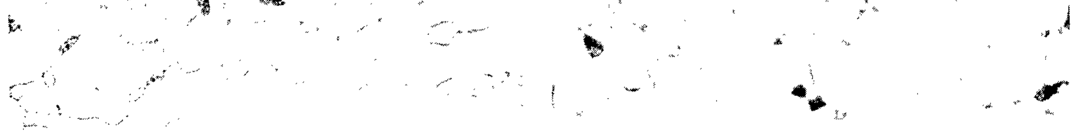


Figure 1—Stress-corrosion cracking in fittings manufactured from 17-7 ph stainless steel etched electrolytically in a 10-percent oxalic acid solution, X500.

eliminate this susceptibility, the authors initiated a limited investigation aimed at accomplishing this objective without materially affecting the strength of the material under test. Hardening induced by cold working is known to improve the resistance of these steels to stress-corrosion. However, it is extremely difficult to form or machine hardened steel into the shapes required in spacecraft; therefore it is usual to employ a thermal hardening treatment following the forming operations. For this reason, the approach taken in this investigation was aimed at developing an improved thermal treatment for the 17-7 ph alloy. This investigation was conducted from one sheet of material of the following chemical composition: 17.4% Cr, 7.26% Ni, 1.12% Al, 0.42% Mn, 0.34% Si.

HEAT TREATMENT

ss

The hardening of steels by heating and cooling phenomena depends on the transformation of austenite to martensite. The martensite (Ms) transformation temperatures for semi-austentic stainless ph steels are sub-zero; the commercial heat treatments (Reference 2) developed for 17-7 ph stainless steel and listed in Table 1(treatments B and C) use austenite conditioning for the express purpose of raising the Ms temperature to near-room temperature. These conditioning temperatures promote chromium-carbide precipitation at the grain boundaries with a consequent loss in stress corrosion resistance. In Reference 3, it is shown that the amount of carbon precipitated at the grain boundaries decreases as the austenite conditioning temperature increases. Therefore, the 1750°F austenite conditioning temperature used in treatment C should produce fewer precipitated carbides than in the 1400°F used in treatment B. In view of these facts and after examining available literature (References 1 through 4), the authors concluded that a direct sub-zero treatment following the solution anneal would induce martensitic transformation in the 17-7 ph alloy without precipitating chromium carbides. Using standard commercially available 17-7 sheet (0.63-inch thick), they gave the samples the three treatments listed in Table 1. They made hardness measurements, to obtain a preliminary idea of the effectiveness of the treatment.

Table 1

Treatments.

Treatment identification	Solution Annealing temperature (°F)	Austenite conditioning temperature (°F)	Sub-zero transformation	Aging temperature (°F)	Percentage of carbon in the form of precipitated carbides	Percentage of retained austenite
B	1950	1400	None	950	0.062	10.0
C	1950	1750	Dry ice	950	0.044	15.0
D	1950	None	Dry ice	950	0.006	26.0

Notes: 1. Values for the percentage of carbon in precipitated carbides were obtained from Reference 3. The value used for the experimental treatment D was considered to be the same as the value for material that was solution-annealed only.
2. The temperatures were within ± 10°F of reported values.

The commercial practices listed in Reference 2 were adopted for the following processes: heat-treating temperatures, holding times, and cooling methods for the commercial treatments B and C; and the solution annealing and aging practices used for the experimental treatment D. The transformation for treatment D in which test samples were held overnight in dry ice was essentially isothermal, taking 8 hours to complete. While the isothermal transformation was not fully investigated, it was ascertained that hardening failed to occur when the samples were placed in liquid nitrogen rather than in dry ice. However, when the samples were warmed up from liquid-nitrogen to dry-ice temperature, hardening occurred.

MICROSTRUCTURE

Figures 2 and 3 show the results of metallographic examinations of samples given the commercial treatments B and C and the experimental treatment D. Samples representing the commercial treatments (Figure 2) exhibited significant carbide formation in the grain boundaries in marked contrast to the structures obtained with treatment D. The oxalic-acid etching applied to the samples of Figure 3 followed the American Society for Testing Materials (ASTM) procedure (Reference 5) recommended in evaluating the susceptibility of stainless alloys to stress corrosion. This procedure demonstrated the presence or absence of carbides (the etchant attacks carbides). When excessive carbides are present in the grain boundaries, the structure has a ditched appearance; the amount of grain-boundary ditching provides a measure of alloy susceptibility to intergranular corrosion failure. Samples given treatment B—the most widely used commercial treatment—exhibited the severest amount of grain-boundary ditching. Samples given commercial treatment C exhibited less ditching, while samples given the experimental treatment D exhibited essentially no ditching (this indicated the absence of precipitated carbides).

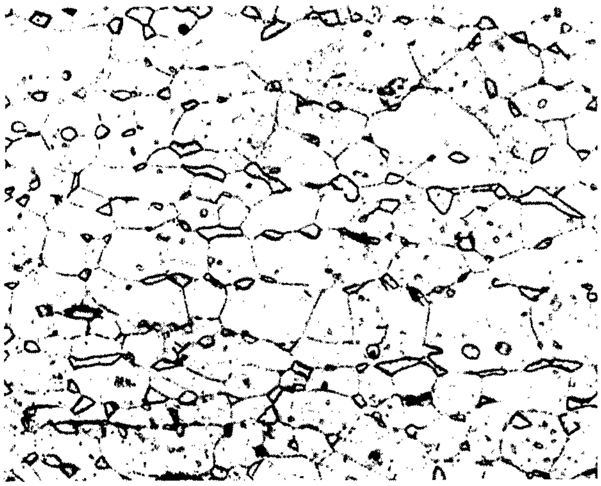
Using Cr $K\alpha$ radiation, samples of each treatment were measured for retained austenite. As was expected, the amount of retained austenite (Table 1) was greater in the experimental heat-treatment D samples.

MECHANICAL TESTS

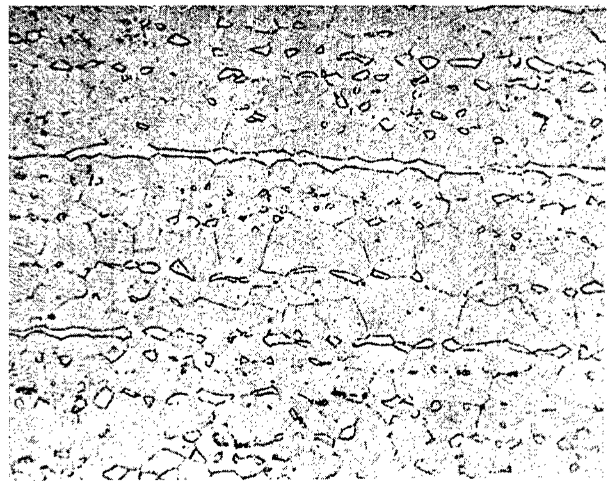
Table 2 presents hardness and tensile data for each of the heat treatments. While commercial treatments B and C yield comparable ultimate tensile properties, experimental treatment D reveals a drop in yield strength with a beneficial increase in ductility, especially in the transverse direction.

STRESS-CORROSION

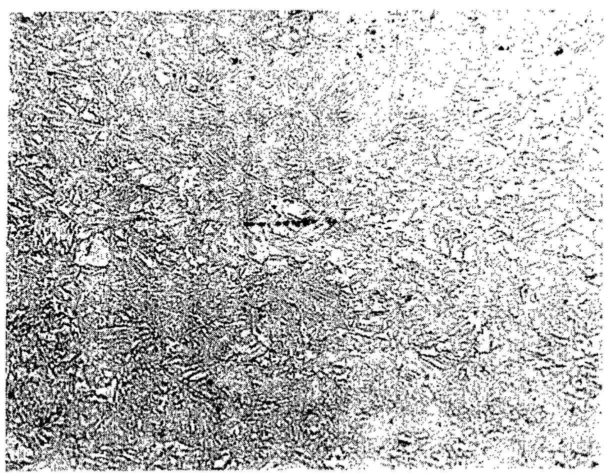
Accelerated stress-corrosion tests were conducted using a boiling (154°C) 42-percent $MgCl_2$ solution in a closed reflux system. The specimens were stressed as a simple beam, as shown in Figure 4. The test fixture permitted the testing of three or four specimens concurrently. Each setup involved specimens of all three heat treatments with the samples randomly positioned in the



(a) after treatment B



(b) after treatment C



(c) after treatment D

Figure 2—Microstructure of 17-7 ph stainless steel given various heat-treatments. Etched electrolytically in a 10-percent oxalic acid solution, X500.

fixture. Varying specimen curvature with rods of different diameter gave the desired level of fiber stressing. The stresses were computed from simple three-point beam-loading formulas.

The test results reported in Table 3 represent the average of several runs made on longitudinal samples at each stress level for the three heat-treat conditions. Components subjected to treatment D evidently last longer under stress-corrosion than do components subjected to treatments B and C. A threshold stress level (104,000 psi) was found below which stress-corrosion did not occur, regardless of the treatment. A transitory stress region between 108,000 and 112,000 psi

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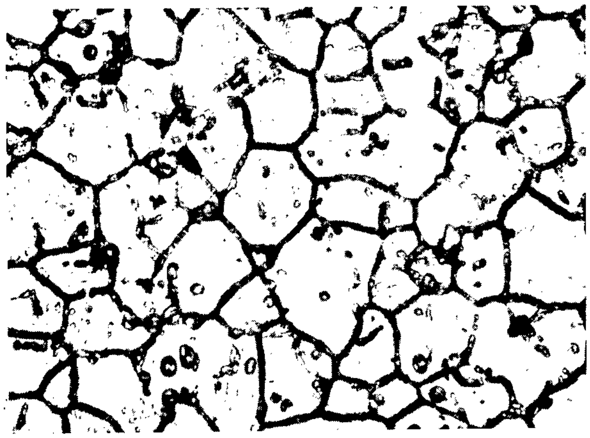
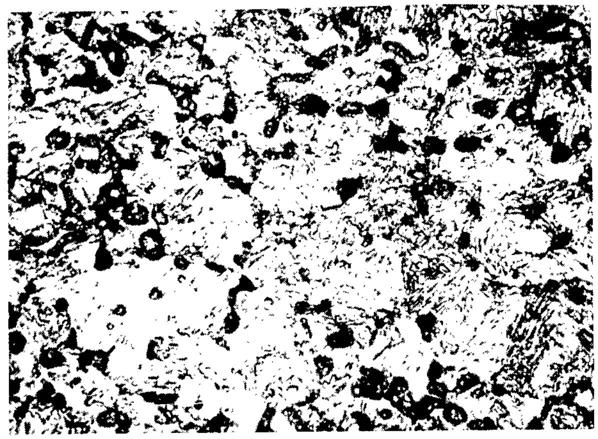
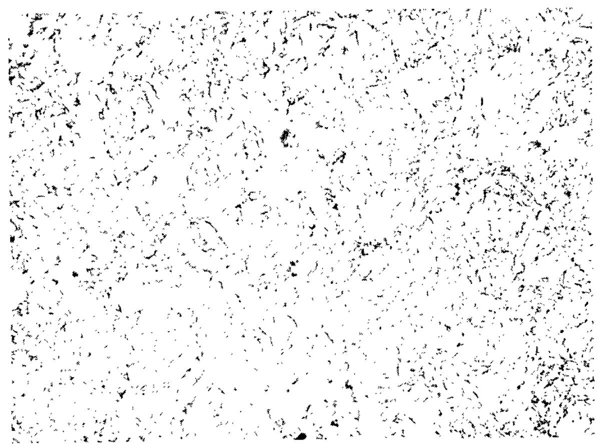


(a) after treatment B



(b) after treatment C



(c) after treatment D

Figure 3—Microstructure of 17-7 stainless steel given various heat-treatments, ASTM oxalic acid etch test (1.5 minutes, 1 amp per cm^2), X500.

Table 2

Average Mechanical Properties.

Treatment identification	Hardness "R_C"	Longitudinal Properties			Transverse Properties		
		Yield strength 0.2 percent offset (psi)	Ultimate tensile strength (psi)	Percent elongation 2 inches	Yield strength 0.2 percent offset (psi)	Ultimate tensile strength (psi)	Percent elongation 2 inches
B	48	202,000	216,000	7.5	195,000	211,000	4.0
C	48	203,000	220,000	8.0	195,000	212,000	4.0
D	45	188,000	213,000	11.0	180,000	210,000	10.0

Notes: 1. Three specimens of each treatment were tested to determine the strength and elongation values. Every specimen was measured for hardness.
2. The strength values listed are within ± 1000 psi., the elongation values are within ± 0.5%, and the hardness values are within ±1 "Rc."

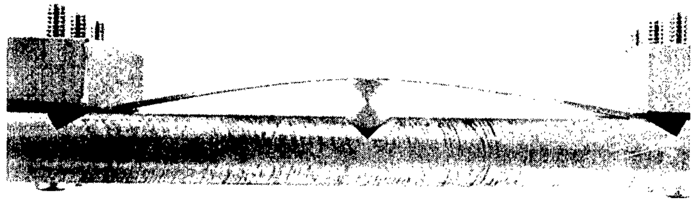


Figure 4—Simple beam method of loading up to four specimens in the same fixture, X1/2.

was found where the test life was from 4 to 120 hours in the case of treatment D and from 2 to 120 hours in the case of treatments B and C. The existence of such a transitory stress range was not surprising, since other investigators (Reference 4) have reported it for other alloys. Table 3 does not give data for this stress range, which was not extensively investigated.

The appearance of the stress-corrosion cracks for the three treatments was not significantly different. The cracks were essentially intergranular.

Table 3

Stress Corrosion Properties.

Treatment	Test life in hours at indicated stresses		
	104,000 psi	119,000 psi	127,000 psi
B	120	1-2	1/2-1
C	120	2-3	1-2
D	120	6-8	3-4

Notes: 1. The tests listed in the second column were terminated after 120.
2. Each reported value represents 3-5 tests.
3. The stress levels are within ± 1000 psi.

SUMMARY

A simplified heat treatment for 17-7 ph stainless steel was found that gives the alloy improved structures by eliminating the bulk of the precipitated carbides from the grain boundaries. Improved resistance to stress corrosion was evident at stresses of 119,000 and 127,000 psi in an accelerated environment of boiling (154°C), 42-percent $MgCl_2$ solution. The high-strength characteristics were not materially affected and the ductility was significantly improved by the new experimental treatment.

end

Goddard Space Flight Center
National Aeronautics and Space Administration
Greenbelt, Maryland, November 20, 1967
492-21-00-01-51

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